5th Quarterly Report – Public Page

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Prepared for: *PHMSA-DOT, National Biodiesel Board, Steel tank Institute, DNV Research and Innovation*

Project Title: *Corrosion and Integrity Management of Biodiesel Pipelines*

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Technical Status

Quarter Five of this research program has seen continued progress in Task 2 (Inhibitors). Data is still being generated based on 30 day exposures of pipe steel samples in inhibited and uninhibited B100 fuel. This is the basis for the matrix of tests to be performed under the guise of Technical Task 2. The next step is to test specimens in blended fuel which will be possible in Q6 due to the recent arrival of non-additive petrodiesel samples. Also associated with the "Inhibitors" is the investigation of the plausibility of making electrochemical measurements to monitor corrosion resistance of pipe steel samples using a micro-electrode technique. The intention was to be able to quickly screen conditions using Linear Polarization Resistance techniques with the micro-electodes. After exhaustive work to establish the initial Cyclic Potentiodynamic Polarization curve on which the LPR measurements can be based, it has been concluded that this route of electrochemical measurement may not be possible in biodiesel fuels. A shift in focus to another electrochemical technique called Electrochemical Impedance Spectroscopy hopes to yield more usable data in the highly resistive fuel blends. Preliminary results are promising as it has been discovered that with a 2-electrode impedance approach the difference in resistivity between blended fuels can be discerned.

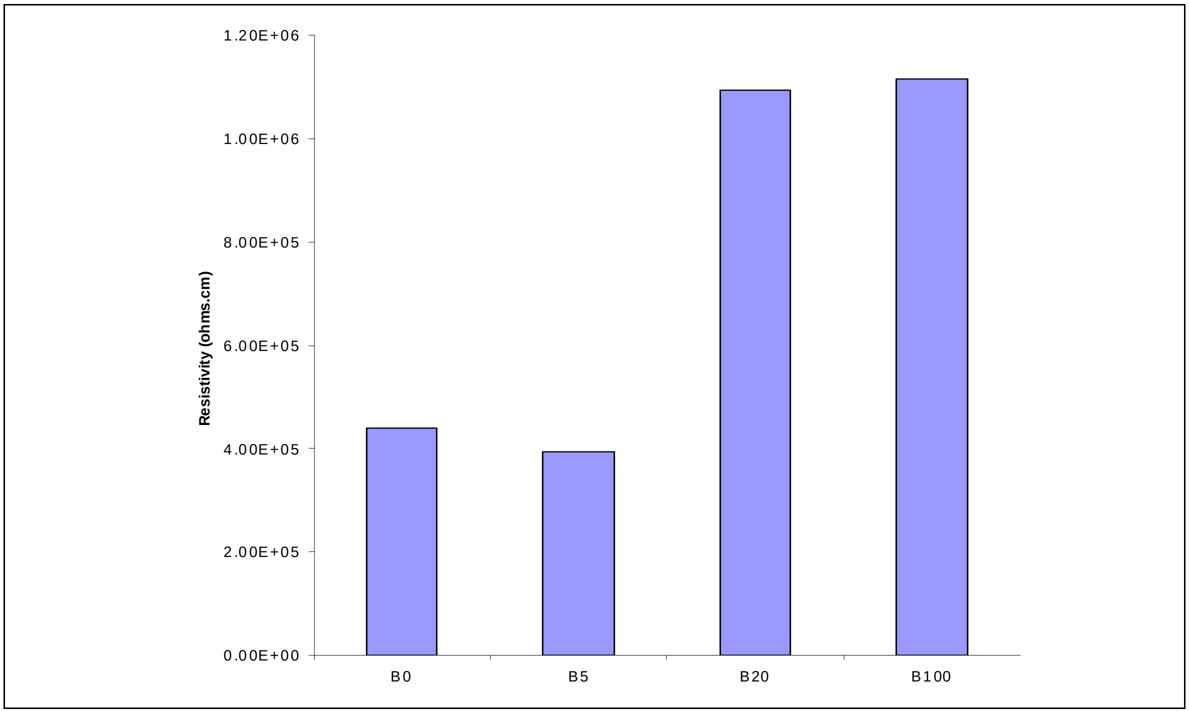

Figure 1. Graph depicting the ability to discern the difference in resistivity of Biodiesel blends using a two electrode impedance approach.

Similar to the intention of using LPR monitoring, EIS screening will also provide a quick screening capability of inhibitors with respect to the corrosion behavior of pipe steel samples. This technique will also be applied to Task 3 in the evaluation of the degradation of Cu-containing alloys (e.g. Brass).

Task 3 involves the electrochemical monitoring of Cu-containing alloy specimens when exposed to biodiesel fuel blends. The corollary to this testing is the documentation of fuel degradation due to the incidence with the Cu-containing alloy. An array of eight test cells similar to what is being used in the inhibitors testing setup has been assembled and the delay in testing is with respect to the availability of a multiplexer for the electrochemical measurements. It is the intention of this work to begin monitoring fuel degradation whether the electrochemical measurements can take place or not. The commencement of the study of corrosion behavior of the Cu-alloy (Alloy 360 Brass) coupons can pick up when ever equipment and viability of the technique becomes available.

For technical tasks 2 and 3, it is still this project's intention to test three different fatty acid saturation levels of biodiesel to address the differing properties of ASTM D 6751 spec fuel. Currently, however, we may be limited by the feedstock made available to us by independent suppliers, outside of the NBB, willing to donate directly to DNV to aide in the progress of our testing program. The National Biodiesel Board has still not yet been able to provide fuel samples for this testing, however Soy-based biodiesel stock has been supplied by independent sources. We have received petrodiesel at the end of Q5 so fuel blends testing will commence in Q6.

Finally, for Task 4, the technical task focused on evolution of physical properties of elastomeric materials, the preliminary testing of the block oven was initiated and it was discovered that new plumbing was required to ensure that the condensation of evaporated fuel would be properly maintained. This construction effort is finished and troubleshooting of the new oven configuration has been completed. Samples of the elastomers have been machined and testing will commence at the end of this week. Delay in projected Q5 progress has been attributed to staffing issues which has now been corrected.

A summary of the three technical tasks is as follows:

Task 2 – Corrosion Inhibition Performance
Task 3 – Integrity of Non-Ferrous Metallic System Components (Cu-alloys)
Task 4 – Integrity of Non-Metallic System Components (Elastomers)

A summation of results and status per task is presenting the following sections.

Task 2 – Corrosion Inhibition Performance

Figure 2. Array of test cells mid-test for Task 2 (inhibitors testing) in biodiesel (an array exactly similar will be used for Task 3 testing).

Each test cell is daisy-chained to a circulating bath that keeps the immersions at 38 +/- 1ºC. 30 day immersions have been documented with neat B100 fuel as well as two dopages of Nalco EC5407A corrosion inhibitor. Samples analyzed for weight loss as well as visually inspected for pit formation on the surfaces. The results from the first sets of immersion tests are tabulated below. These data represent a raw material cut from tank steel provided by STI. Subsequent tests were carried out with samples whose surfaces were 600 grit finish or greater.

Inhibitor	Concentration	visual inspection (# of pits)	average weight change (g)	average corrosion rate (mpy)
N/A	0	0	11.6	1.275
	5 ppm	0	1.45	0.159
Nalco	10 ppm	0	4.85	0.533

Figure 3. typical results from inhibitors testing

The “No inhibitor” testing condition was retested with the new samples to elucidate the effect of surface finish. The results were considerable enough that the Nalco inhibitor in B100 will be retested at both dosages. Tabulated below are the weight loss results for the other two Inhibitors involved in the matrix: A Tolad and a Dupont specified corrosion inhibitor.

Inhibitor	Concentration	visual inspection (# of pits)	average weight change (mg)	average corrosion rate (mpy)
N/A	0	0	0.1	0.011
	3 mg/L	0	0.1	0.011
Tolad	11 mg/L	0	0.1	0.011
	5 ppm	0	0.2	0.022
Dupont	10 ppm	0	0.2	0.022

Figure 4. Corrosion Rate results for test in B100 with Tolad and Dupont corrosion inhibitors.

The second portion of inhibitors testing is the exploratory electrochemical monitoring of corrosion behavior first via a micro-electrode technique and most recently employing a 2-electrode impedance technique. Exhaustive testing using the microelectrode technique including trials where supporting electrolytes were used, such as Tetrabutylammonium Tetrafluoroborate, yielded no consistent results and the conclusion was made to not waste any more time on this methodology. Some documentation of the attempt exists in the Q4 report.

Task 3 – Integrity of Non-Ferrous Metallic System Components (Cu-alloys)

Task 2 has two main objectives which remain unchanged. The first material-related objective is to document the degradation over time of Cu-containing alloys when immersed in the various blends of biodiesel. For this, Alloy 360 will be utilized as a prudent example of common Cu-based alloys which experience regular incidence with the fuel in pump housings and seals. A series of eight additional test cells like pictured in Figure 1 will be allocated to electrochemical monitoring of the Cu-alloy corrosion behavior using a multiplexed potentiostat. The testing has not begun due to unavailability of the aforementioned multiplexer. Upon further consideration of the start of it was a better use of resources and time to hope for the arrival of petrodiesel samples so that a more complete matrix of conditions can be setup simultaneously. This will provide more ease in cataloging of fuel evolution and impedance breakdown which are both time-relative monitoring tasks. Incidentally, Petrodiesel samples have arrived in the last month and the process of switching over the task 2 setup to task 3 testing is being made.

Task 4 – Integrity of Non-Metallic System Components (Elastomers)

For the testing of elastomer materials, a 28 test tube block furnace is used. Each test tube will hold contain one specific experiment in the matrix. Preliminary testing of the block oven pictured below signaled that the hose network needed to be re-plumbed to ensure that the composition of the test fuels will not change due to evaporation over time.

Figure 5. “Medusa”, the 28 vessel block oven used for elastomeric testing in fuel.

Task 5 testing should commence in the beginning of Q5 and data will be available shortly there after.

This project unfortunately has been hindered by one setback after another, but as soon as all of the preliminary struggles are conquered, the data collection should proceed smoothly and regularly moving forward.

Plans for Future Activity

With the acquisition of petrodiesel fuel samples, fuller fields of data will be generated in coming quarters. The testing program was designed to make use of large throughput testing techniques in anticipation of large matrices dictated by several fuel blends and feedstocks. The feedstock variable has been all but eliminated from the dimensions of the testing matrix but thankfully petrodiesel samples will still allow for testing in various biodiesel blends. Q6 will all three technical tasks generating data.